

PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Docket No: Q78309

Yutaka TOSAKI, *et al.*

Appln. No.: 10/701,496

Group Art Unit: 1771

Confirmation No.: 7626

Examiner: Daniel R. ZIRKER

Filed: November 6, 2003

For: PRESSURE-SENSITIVE ADHESIVE TAPE OR SHEET

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Yutaka TOSAKI, hereby declare and state:

THAT I am a citizen of Japan;

THAT I graduated from Kobe University, Faculty of Engineering, Department of Industrial Chemistry, in March of 1986;

THAT I have been employed by Nitto Denko Corporation since April of 1986, where I have been engaged, from 1986 to the present, in research and development regarding pressure-sensitive adhesive tape; and

THAT I am familiar with the prosecution of the above-identified U.S. patent application, including the final Office Action mailed March 15, 2007, containing a rejection of Claims 1-7 under 35 U.S.C. § 102(b) as allegedly being anticipated by U.S. Patent No. 5,571,617 to Coopridier *et al.*

I have conducted the following experiment to show that Cooprider does not inherently disclose an amount of a component corresponding to claimed component (B) present in a surface portion of the PSA layer, i.e., in a portion of the PSA layer within the range of up to 3 nm inward from an outer face of the PSA layer, falling within the claimed range of 0.1 to 3 parts by weight based on 100 parts by weight of the whole of the monomer components constituting the acrylic polymer (A) that forms the surface portion of the pressure-sensitive adhesive layer.

(1) Reproduction Experiment of the Examples in the Cited Reference

(1-1) Reproduction experiment of Cooprider et al. (U.S. Patent No. 5,571,617) and analysis of the ratio of sulfur element on the surface

As the example to be reproduced, Example 8 was chosen. The reason why this composition has been chosen lies in the fact that ammonium lauryl sulfate, which is mentioned as an example in the present application, is used as the emulsifier for Cooprider. The use amount thereof is 1.74 parts ( $5.8 \text{ g} \times 30\% \text{ base} = 1.74 \text{ g}$ ) (0.48 parts by weight relative to 100 parts by weight of an acrylic copolymer: iOA + AA = 364 parts by weight,  $1.74/364 \times 100 = 0.48$  parts by weight). Moreover, the other examples of Cooprider are considered to basically demonstrate a similar trend to the example that has been traced on the present occasion.

The most significant difference between the cited reference and the present application is whether a hydrophilic polymer is added during or after polymerization. The influence of the polymerization method of Cooprider on the amount of surface-concentrated emulsifier has been confirmed.

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Example 8 of Cooprider et al.

Material	Weight (g)	Weight of solid content (g)	Maker	Name of Commercial Product
Deionized water	650			
Ammonium lauryl sulfate	5.8 (30%)	1.74	Stepan Company	Polystep B7
Polyacrylic acid (MW: 190,000)	7 (25%)	1.75	Rohm and Haas Company	Acumer IS-30
Ammonium hydroxide	1.4			
Acrylic acid	14	14		
Isooctyl acrylate	350	350		
Active bis(4-tert-butylcyclohexyl) peroxydicarbonate	1.1 (95%)	1.045	ACZO Chemicals, Inc	Perkadox 16N

According to the aforementioned example of the Cooprider and with the following formulation, a reproduction experiment was carried out. In principle, the chemical formulations were perfectly the same except polyacrylic acid. Since the same grade (Mw: 190,000) of polyacrylic acid was difficult to obtain from temporal condition, polymerization was carried out by obtaining two grades of polyacrylic acid with different molecular weights. As shown in the table below, the two grades used had molecular weights of 150,000 and 250,000 in comparison to the molecular weight of 190,000 in the cited reference. Thus one grade is slightly lower and the other is slightly higher than that of the cited reference in molecular weight. By considering these results, the result on the grade of 190,000 molecular weight of the cited reference can easily be anticipated. In addition, in the cited reference, the concentrated ammonium hydroxide is stoichiometrically added as a base that gives rise to ammonium polyacrylate. In Example 6, 2.8 g is added relative to 14 g of polyacrylic acid. Accordingly, in Example 8, concentrated ammonium hydroxide was added by 1.4 g since the amount of polyacrylic acid was 7 g.

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Reproduction Experiment 1. (Example 8 of Cooprider et al.)

Material	Weight (g)	Weight of solid content (g)	Maker	Name of Commercial Product
Deionized water	650			
Ammonium lauryl sulfate	7.25 (24%)	1.74	Kao Co.	Emal AD-25R
Polyacrylic acid (MW: 150,000)	7 (25%)	1.75	Nihon Jun-yaku Co., Ltd.	JURYMER AC-10LHP
Ammonium hydroxide	1.4			
Acrylic acid	14	14		
Isooctyl acrylate	350	350		
Active bis(4-tert-butylcyclohexyl) peroxydicarbonate	1.1 (95%)	1.045	NOF Co.	PEROYL TCP

Reproduction Experiment 2. (Example 8 of Cooprider et al.)

Material	Weight (g)	Weight of solid content (g)	Maker	Name of Commercial Product
Deionized water	650			
Ammonium lauryl sulfate	7.25 (24%)	1.74	Kao Co.	Emal AD-25R
Polyacrylic acid (MW: 250,000)	7 (25%)	1.75	Wako Pure Chemical Industries, Ltd.	
Ammonium hydroxide	1.4			
Acrylic acid	14	14		
Isooctyl acrylate	350	350		
Active bis(4-tert-butylcyclohexyl) peroxydicarbonate	1.1 (95%)	1.045	NOF Co.	PEROYL TCP

The polymerization methods adopted in the aforementioned reproduction experiments 1 and 2 were performed according to the detailed descriptions set forth in Example 8 of the cited reference, i.e., in Example 6. Further, production of pressure-sensitive adhesive tape was performed by coating and drying each composition on one side of a Japanese paper (substrate) with a basis weight of 30 g/m<sup>2</sup> in such a manner to give a thickness of 27  $\mu$ m after drying as set forth in Example 3 of the present application.

The ratio of sulfur element on the surface in the pressure-sensitive adhesive tape was measured and derived by the ESCA method as set forth in the present application. According to this method, the elementary ratio in a surface portion of the pressure-sensitive adhesive layer with the range up to 3 nm inward from the outer face of the pressure-sensitive adhesive layer can be obtained. Since the formulation on the present occasion only contains the emulsifier as the sulfur-containing material, the ratio of sulfur element corresponds to the amount of the emulsifier.

The result is shown in Table 1.

Table 1. Result of measurement of elementary ratios (in atomic %)

	C	N	O	Si	S
Reproduction Experiment 1	82.35	1.0	15.2	0.95	0.5
Reproduction Experiment 2	83.0	2.0	14.25	1.05	0.4

From the result in the table above, the sulfur element ratios in reproduction experiments 1 and 2 were determined to be 0.5 and 0.4 atomic %, respectively.

(1-2) Reproduction experiment 1: Result and discussion on the derivation of the surface sulfur element (amount of emulsifier) for the case where polyacrylic acid (Mw: 150,000) is used.

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From the formulation in the cited reference, the ratio of sulfur element can be obtained as shown in the following table with the assumption of uniform distribution of elements.

Table 2. Number of elements and molecular weight of each material

	C	H	O	N	S	Mw
Ammonium lauryl sulfate	12	29	4	1	1	283
Polyacrylic acid (MW: 150,000)	6249	8332	4166			150000
Conc. Ammonium hydroxide		5	1	1		35
Acrylic acid	3	4	2			72
Isooctyl acrylate	11	20	2			184
Initiator (Perkadox 16N)	22	38	6			398

Table 3. Formulated number of parts and molar number for each material (derived from the parts and Mw of Table 2.)

	Part	Molar number
Ammonium lauryl sulfate	1.74	0.0061
Polyacrylic acid (MW: 150,000)	1.75	0.000012
Conc. Ammonium hydroxide	1.4	0.0400
Acrylic acid	14	0.1944
Isooctyl acrylate	350	1.9022
Initiator (Perkadox 16N)	1.045	0.0026

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Table 4. Number of element of each material (calculated from the number of elements in Table 2 and the molar number in Table 3)

	C	H*	O	N	S
Ammonium lauryl sulfate	0.074		0.025	0.006	0.006
Polyacrylic acid (MW: 150,000)	0.073		0.049	0.000	0.000
Conc. Ammonium hydroxide	0.000		0.040	0.040	0.000
Acrylic acid	0.583		0.389	0.000	0.000
Isooctyl acrylate	20.924		3.804	0.000	0.000
Initiator (Perkadox 16N)	0.058		0.016	0.000	0.000
Number of elements	21.7	0.0	4.3	0.046	0.006
26.1 in total					
Ratio of elements, converted to 100%	83.2	0.0	16.6	0.2	0.02357

\*Note: Hydrogen cannot be measured by ESCA.

From the above calculation result, and with the assumption of uniform distribution of element in the entire thickness direction of the pressure-sensitive adhesive layer, the ratio of sulfur element was determined to be 0.02357%.

In contrast thereto, the ratio of sulfur element was 0.5 atomic % as shown in Table 1 above. By using this value and calculating the number of parts according to the steps reversal to those of Tables 2 to 4 above, ammonium lauryl sulfate was derived to be 40.4 parts by weight relative to 364 parts by weight of monomer (i.e., 11.1 parts by weight relative to 100 parts by weight of monomer) (Tables 5 and 6).

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Namely, it has proved that the emulsifier existed in the actual surface portion at the ratio of 11.1 parts by weight in comparison to 0.48 parts by weight when the emulsifier is assumed to be distributed uniformly over the entire thickness direction of the pressure-sensitive adhesive layer. This concentration difference is by a factor of as large as about 23, obviously deviating from the range of from 0.1 to 3 parts by weight defined by the present application.

Table 5. Derivation of the amount of ammonium lauryl sulfate required to give the ratio of sulfur element (in Table 6) of 0.5

	Part	Molar number
Ammonium lauryl sulfate	40.4	0.1428
Polyacrylic acid (MW: 150,000)	1.75	0.000012
Conc. Ammonium hydroxide	1.4	0.0400
Acrylic acid	14	0.1944
Isooctyl acrylate	350	1.9022
Initiator (Perkadox 16N)	1.045	0.0026

Table 6.

	C	H	O	N	S
Ammonium lauryl sulfate	1.713		0.571	0.143	0.143
Polyacrylic acid (MW: 150,000)	0.073		0.049	0.000	0.000
Conc. Ammonium hydroxide	0.000		0.040	0.040	0.000
Acrylic acid	0.583		0.389	0.000	0.000
Isooctyl acrylate	20.924		3.804	0.000	0.000
Initiator (Perkadox 16N)	0.058		0.016	0.000	0.000
Number of elements	23.4	0.0	4.9	0.183	0.143
28.5 in total					
Ratio of elements, converted to 100%	81.8	0.0	17.1	0.6	0.50011



(1-3) Reproduction experiment 2: Result and discussion on the derivation of the surface sulfur element (amount of emulsifier) for the case where polyacrylic acid (Mw: 250,000) is used.

From the formulation in the cited reference, the ratio of sulfur element can be obtained as shown in the following table with the assumption of uniform distribution of elements.

Table 7. Number of elements and molecular weight of each material

	C	H	O	N	S	Mw
Ammonium lauryl sulfate	12	29	4	1	1	283
Polyacrylic acid (MW: 250,000)	10416	13888	6944			250000
Conc. Ammonium hydroxide		5	1	1		35
Acrylic acid	3	4	2			72
Isocetyl acrylate	11	20	2			184
Initiator (Perkadox 16N)	22	38	6			398

Table 8. Number of the formulated parts and the molar number of each material (calculated from the number of parts and the molecular weight in Table 7)

	Part	Molar number
Ammonium lauryl sulfate	1.74	0.0061
Polyacrylic acid (MW: 250,000)	1.75	0.000007
Conc. Ammonium hydroxide	1.4	0.0400
Acrylic acid	14	0.1944
Isocetyl acrylate	350	1.9022
Initiator (Perkadox 16N)	1.045	0.0026

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Table 9. Number of elements of each material (calculated from the number of elements in Table 7 and the molar number in Table 8)

	C	H	O	N	S
Ammonium lauryl sulfate	0.074		0.025	0.006	0.006
Polyacrylic acid (MW: 250,000)	0.073		0.049	0.000	0.000
Conc. Ammonium hydroxide	0.000		0.040	0.040	0.000
Acrylic acid	0.583		0.389	0.000	0.000
Isooctyl acrylate	20.924		3.804	0.000	0.000
Initiator (Perkadox 16N)	0.058		0.016	0.000	0.000
Number of elements	21.7	0.0	4.3	0.046	0.006
26.1 in total					
Ratio of elements, converted to 100%	83.2	0.0	16.6	0.2	0.02357

From the calculation result heretofore, the ratio of sulfur element was determined to be 0.02357% when the elements were assumed to exist uniformly over the entire thickness of the pressure-sensitive adhesive layer.

In contrast thereto, the ratio of sulfur element was 0.4 atomic % as shown in Table 1 above. By using this value and calculating the number of parts according to the steps reversal to those of Tables 5 to 7 above, ammonium lauryl sulfate was derived to be 31.7 parts by weight relative to 364 parts by weight of monomer (i.e., 8.7 parts by weight relative to 100 parts by weight of monomer) (Tables 10 and 11).

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Namely, it has proved that the emulsifier existed in the actual surface portion at the ratio of 8.7 parts by weight which is about 18 times larger than 0.48 parts by weight, the value when the emulsifier is assumed to distribute uniformly in the entire thickness direction of the pressure-sensitive adhesive layer. This concentration difference obviously deviates from the range of from 0.1 to 3 parts by weight defined by the present invention.

Table 10. Calculation of the amount of ammonium lauryl sulfate required to make the ratio of sulfur element in Table 11 0.4

	Part	Molar number
Ammonium lauryl sulfate	31.7	0.1120
Polyacrylic acid (MW: 250,000)	1.75	0.000007
Conc. Ammonium hydroxide	1.4	0.0400
Acrylic acid	14	0.1944
Isooctyl acrylate	350	1.9022
Initiator (Perkadox 16N)	1.045	0.0026

Table 11.

	C	H	O	N	S
Ammonium lauryl sulfate	1.344		0.448	0.112	0.112
Polyacrylic acid (MW: 250,000)	0.073		0.049	0.000	0.000
Conc. Ammonium hydroxide	0.000		0.040	0.040	0.000
Acrylic acid	0.583		0.389	0.000	0.000
Isooctyl acrylate	20.924		3.804	0.000	0.000
Initiator (Perkadox 16N)	0.058		0.016	0.000	0.000
Number of elements	23.0	0.0	4.7	0.152	0.112
28.0 in total					
Ratio of elements, converted to 100%	82.1	0.0	17.0	0.5	0.40017

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(2) Conclusion

From the result of the aforementioned reproduction experiments, it is obvious that the cited reference of Coopridier clearly deviates from the range defined by the present application. Namely, with respect to the description set forth in Claim 1 of the present application, "in a surface portion of the pressure-sensitive adhesive layer within the range of up to 3 nm inward from the outer face of the pressure-sensitive adhesive layer, an anionic emulsifier containing a sulfur atom is contained in a proportion of from 0.1 to 3 parts by weight based on 100 parts by weight of the whole of the monomer components constituting the acrylic polymer," the anionic emulsifier non-uniformly exists in from 8.7 to 11.1 parts by weight at the surface portion, thus largely deviating from the range claimed in the present application. Hence, it can be concluded that the present invention cannot be anticipated by the cited reference, Coopridier.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: July 9, 2007

Yutaka TOSAKI  
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